A Scanning Photometer

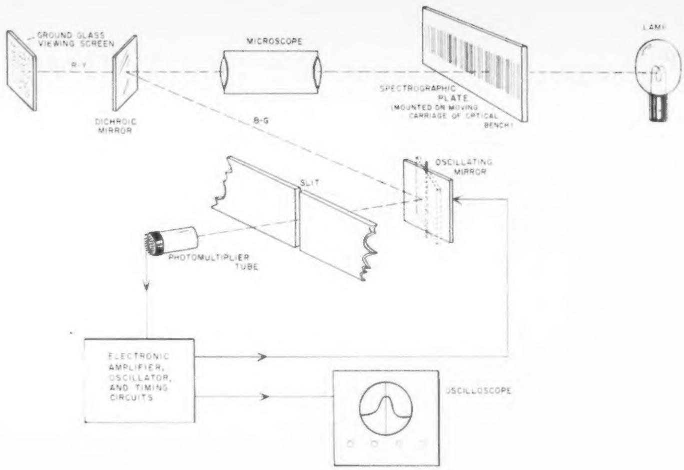
FOR READING SPECTROGRAPHIC PLATES

The National Bureau of Standards has developed an improved scanning photometer to determine wavelengths of spectral lines on a spectrographic plate easily, rapidly, and accurately. The instrument, devised by M. L. Kuder of the electronic instrumentation laboratory, optically scans a 0.5-mm-wide portion of the plate and then presents, on an oscilloscope tube, a curve of spectral line density versus wavelength. The instrument was developed for the spectroscopic laboratory to help automate the processing of a large volume of spectrographic plates.

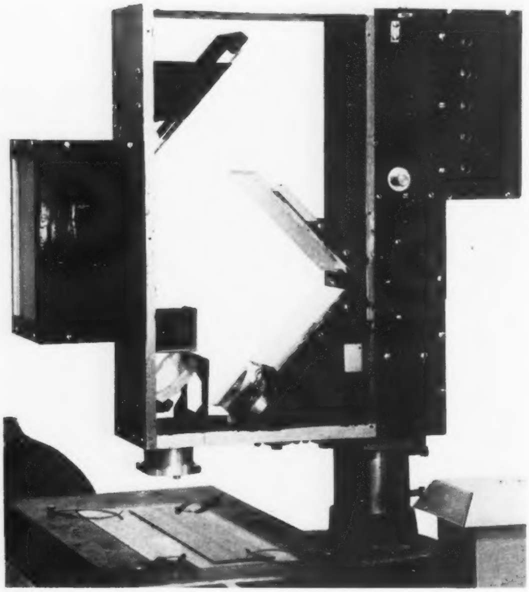
The plate is mounted on an optical bench, and any part of the spectrum can be scanned by manually shifting the position of the moving carriage. A fixed fiducial line on the face of the oscilloscope tube aids in locating the exact center of each spectral line. Wavelength can then be determined by measuring the displacement of the plate from some known starting point. Not only is the electronic equipment unusually stable, but it is also sensitive enough to detect many lines in the hyperfine structure that are too faint to be readily seen by the human eye.

For many years, the conventional method for reading spectrographic plates has been microscopic examination of the spectral lines. The plate is usually mounted on a traveling table with vernier scales to indicate displacement from a fixed point. Through a fixed microscope, an operator locates the center of a line, as well as he is able, and reads and records a value for displacement. This value is later converted to wavelength. As this procedure is tedious, time-consuming, and susceptible to simple error due to eyestrain, the Bureau has investigated methods to improve the procedure. The present instrument is based on an electronic scanning device, developed elsewhere,[1] that scans the plate with a rotating prism and presents the density versus wavelength curve as an oscilloscope trace.

In the Bureau's improved instrument, the plate is illuminated on one side; on the other side a 10-power microscope is trained on a limited area of the spectrum. Immediately behind the microscope is a dichroic mirror that splits the image into two components: one, red-yellow and the other, blue-green. The red-yellow image is reflected to a ground glass viewing screen, which provides the operator with a visual check of the portion of the spectrum being scanned. The blue-green image is transmitted to a vibrating mirror, which reflects an oscillating image through a narrow slit to an end-on photomultiplier tube. The vibrating mir-



Right: **Schematic of improved scanning photometer. The instrument scans a spectrographic plate and presents on an oscilloscope tube a curve of spectral line density versus wavelength.** ***Below:*** **Spectrographic plate is laid flat on carriage at bottom of photograph, and is illuminated from underneath. Immediately above plate is microscope and dichroic mirror. To right of dichroic mirror is oscillating mirror that provides scanning action. Box at left holds ground glass viewing screen; box at upper right contains phototube.**



ror provides the scanning action of the system, and the phototube "sees" only a 13-μ width of the spectrum at any give time, although a total width of 0.5 mm is being scanned.

The electrical output of the tube is fed to an electronic network with appropriate amplifying, timing, and positioning circuits. The resulting density-versus-wavelength curve is presented together with a fiducial line on the face of an oscilloscope tube. As the horizontal sweep of the oscilloscope (corresponding to the scan along the spectrographic plate), the vibrating mirror, and the fiducial line generator are all driven by the same oscillator, and the phase relation between the three is constant, the display holds steady and is completely free from drift. The only time this curve moves is when the carriage carrying the plate is manually shifted on the optical bench to examine another portion of the spectrum. The fiducial line, of course, is steady.

Although only one phototube has been mentioned, the instrument actually has five such tubes, one to detect each of the five separate spectra that are usually photographed side by side on a single plate. The operator can examine any one of the spectra simply by turning a 5-position switch. Two of the spectra are standards (such as Hg 198) whose most prominent lines have previously been measured precisely on an interferometer. The presence of these lines on the plate gives the operator a set of calibrated points between which he can interpolate to determine accurately the wavelengths of the lines appearing in the three unknown spectra.

Connected with the moving carriage is equipment to convert displacement into a digital value of wavelength. This value, together with a visual estimate of line density, can be recorded on a punched card through simple key controls whenever the operator has located the center of a line. The information is then available for analysis or printout by a high-speed electronic computer.

Plans are now under way to develop equipment that will locate lines and measure their densities electronically. When this equipment is completed, the reading of spectrographic plates will be entirely automatic.

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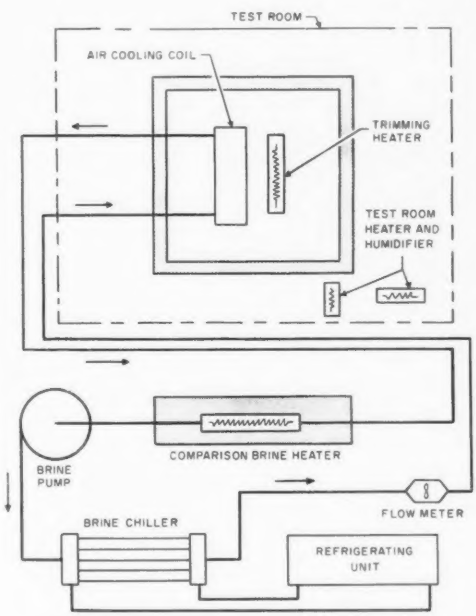
[1] **A photoelectric setting device for a spectrum plate comparator,** ***by F. S. Tompkins and M. Fred, J. Opt. Soc.*** **41,** ***641 (Sept. 1951).***

Standard Method for Measuring Cooling Loads of Refrigerated Trailers

A "HEAT SINK method" for measuring cooling loads of refrigerated trailers and refrigerated structures has been developed by the Bureau in a program sponsored by the U.S. Department of Agriculture, the Quartermaster Research and Engineering Command of the Department of the Army, and the Truck-Trailer Manufacturers Association. The method was developed by P. R. Achenbach, C. W. Phillips, and W. F. Goddard of the refrigeration laboratory.

The heat sink method [1] is part of a Standard Testing and Rating Method for Refrigerated Trailers with Respect to Cooling Load, Weight Gain and Air Leakage, recently adopted by the Truck-Trailer Manufacturers Association. Previously there had been no standard cooling load measurement method for refrigerated trailers.

The standard testing and rating method will enable the trailer manufacturer to establish a cooling load rating for each of his models. The purchaser, knowing these ratings, and giving consideration also to the products being carried, the length of the haul, and other factors, will be aided in selecting the size of refrigerating unit best suited to his needs. He should be able to avoid selecting equipment either inadequate to deliver the food cargo in unspoiled condition, or unnecessarily costly for the job.



Heat sink apparatus developed for measuring the cooling loads of refrigerated trailers. The heat sink method is part of a Standard Testing and Rating Method for Refrigerated Trailers recently adopted by the Truck-Trailer Manufacturers Association.

The standard method as adopted applies to refrigerated trailers of any length used for transporting frozen food or other materials at cargo space temperatures well below 32° F. The standard describes a procedure for measuring trailer cooling loads in the laboratory as a basis for rating the trailers. Values obtained in this way represent the cooling loads under typical road speeds. The effect of ram air pressure on the front of a moving trailer is simulated in the laboratory during the rating test by means of a pressurized plenum.

Two simultaneous procedures—both employing the "heat sink" apparatus— are used in the rating test in the laboratory to determine the cooling load under the standard test conditions of 0° F temperature in the trailer and 100° F dry bulb temperature and 50 percent relative humidity in the test room. For purposes of both procedures, chilled brine is pumped continuously through a closed loop which includes an air cooling coil inside the trailer, a comparison brine heater outside, a brine pump, a brine chiller, and an electronic flowmeter.

Under the first of these procedures, the comparison method, the cooling load is measured by a comparison between the temperature rise of brine in the trailer and in the external comparison heater. Under the second procedure, the cooling load is determined from the temperature rise of the brine in the trailer and the mass flow of brine as measured by the flowmeter. The results from the two procedures must agree within 5 percent for a given test to be acceptable for rating purposes. The rated cooling load is the average of values obtained by the two methods, expressed to the nearest 500 Btu/hr.

The heat sink apparatus serves both to produce the desired low temperature inside the trailer and to measure the rate of heat transfer into the vehicle. As developed at the Bureau, the apparatus consists of three electrically-driven two-speed compressors, with water-cooled condensers, a water-cooling tower, two brine chillers, the brine pump, the external comparison brine heater, a cooling coil inside the trailer, and the electronic flowmeter and other suitable measuring instruments.

Comparison Method

The comparison brine heater consists of a vaporproofed, insulated box containing an electric immersion heater inserted in the brine line which passes through the box as part of its circuit. The electric energy consumed by the heater is measured with a calibrated watthour meter.

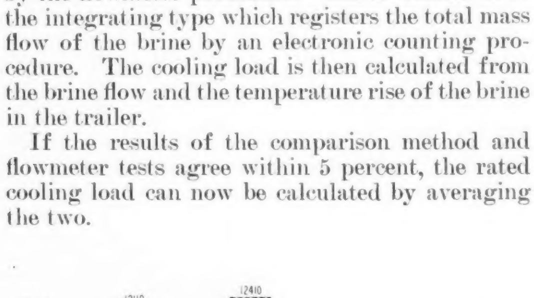
Since the brine flow rate in all parts of the closed brine circuit is the same, the ratio of temperature rise of the brine in the air cooling coil in the trailer to the temperature rise in the external comparison brine heater is equal to the ratio of the heat absorbed in the coil to that absorbed in the heater. (Changes in specific heat and the slight heat leakage are small enough to be neglected in these tests.)

After corrections for auxiliary heat sources inside the trailer, the above ratio is used to calculate the cooling load resulting from heat transferred through the trailer body by transmission, air leakage, and frost accumulation.

Flowmeter Method

At the same time the closed brine circuit is providing data for measurement of the cooling load by the flowmeter procedure. The flowmeter is of the integrating type which registers the total mass flow of the brine by an electronic counting procedure. The cooling load is then calculated from the brine flow and the temperature rise of the brine in the trailer.

If the results of the comparison method and flowmeter tests agree within 5 percent, the rated cooling load can now be calculated by averaging the two.



Observed laboratory cooling loads (before application of the pressurized plenum), and the extrapolated road cooling loads of four commercial refrigerated trailers. The graph shows the computed values of cooling loads resulting from transmission and air leakage in each case. The pressurized plenum was added to the laboratory apparatus to simulate approximately the air leakage conditions during road operation.

The brine used for the tests is methylene chloride, selected because of its favorable viscosity, and its stability of density and specific heat in the working temperature range, and because it is not excessively toxic or flammable.

Road Tests

In order to study the effects of air leakage and other factors encountered on the road, four commercial trailers were tested on the Ohio Turnpike as part of the program. A modified heat sink apparatus was used to measure cooling loads during the road tests. Comparison of test results observed on the road and in the laboratory made it possible to simulate the road conditions of air leakage in the laboratory.

On the road, the flow rate of the brine was measured with the electronic flowmeter. Then the specific heat and the flow rate of the brine and its temperature change in the trailer cooling coil were used to calculate the cooling loads of the trailers. A comparison electric brine heater of reduced capacity was used at intervals to monitor the performance of the flowmeter.

Test Specimens and Equipment

Seven refrigerated trailers were used for the studies which served as a basis for drafting the Standard Testing and Rating Method. Four were commercial and three were military vehicles. The military vehicles were 7½-ton, 21-ft, single-axle trailers manufactured to U.S. Army specifications. The four commercial trailers were approximately 35 ft long. Their empty weights varied from 9,722 to 13,510 lb. All were designed for low-temperature service with 4 to 6 in. of glass fiber or rigid plastic insulation in floors, walls, and roofs.

The room used for the laboratory tests at the Bureau was 60 ft long, 14 ft wide, and 16 ft high. It had insulated walls and roof and a concrete floor. A sliding overhead door permitted movement of semitrailers into the space.

Weight Gain and Air Leakage

The Standard Testing and Rating Method, as adopted by the Truck-Trailer Manufacturers Association, provides that a Standard Weight Gain Rating and a Standard Air Leakage Rating should be determined for each trailer tested but need not be published in order to publish the Standard Cooling Load Rating. The weight gain and air leakage ratings will be useful to the manufacturer in future design and construction of trailers.

In rating trailers by the standard, the rated weight gain (caused by condensation and freezing

One of four commercial trailers used in developing the Standard Method for Measuring the Cooling Loads of Refrigerated Trailers.

of infiltrated moisture) is the average weight gain rate in pounds per hour for the final 24 hr of the test. It is expressed to the nearest 0.1 lb/hr.

For the air leakage test in the laboratory, air is forced into the trailer, and the amount of air required to maintain a positive pressure of 0.1 in. water gage in the trailer is measured. The rated air leakage is expressed to the nearest 0.5 ft ^{3}/min, in accordance with the adopted standard method.

The results of the tests at the Bureau indicate that smaller refrigerating units could be used if air leakage of trailers could be eliminated, or alternately, less insulation might be required if air leakage were significantly reduced. It is also probable that deterioration of trailer bodies would proceed more slowly if moisture could be kept out of insulation spaces.

[1] *For more detail on the heat sink apparatus, see* Heat sink method for measuring the cooling loads of refrigerated structures, *by P. R. Achenbach and C. W. Phillips, Proceedings Xth International Congress of Refrigeration, 1959.*

Committee Reports on National Bureau of Standards

SECRETARY of Commerce Frederick H. Mueller on April 8 released the report of a Special Advisory Committee of the National Academy of Sciences on "The Role of the Department of Commerce in Science and Technology".

"Science and technology", the Committee asserted, "are playing an increasingly larger part in the industrial and business activities of the nation." The report affirmed the importance and relevance of the Department in this modern role and appraised its programs to insure that the Department fulfills "its responsibilities in the progress of the nation's science and technology".

The Committee recommended the establishment of an Office of Assistant Secretary for Science and Technology to be filled by an official "with professional and management experience in science and technology", and specific expansions of agency programs in science and technology.

Secretary Mueller designated Under Secretary of Commerce Philip A. Ray, Department representative on the Federal Council for Science and Technology, to initiate prompt measures for appraisal of the report and to propose plans for a course of appropriate implementation.

A special study of the Bureau was made by Committee members Dr. Augustus Kinzel, Vice President for Research, Union Carbide Corp.; and Dr. C. G. Suits, Director of Research, General Electric Co.

Excerpts from the report on NBS follow:

"The Panel chose as consultants Mr. R. W. Larson and Mr. George M. Morrow, men of extensive background in research and development. They have made visits, at our direction, to all laboratory areas of the Bureau and have discussed the work with the Director, Associate Directors, program leaders, and specialists engaged in the programs. Extensive use of members of the Advisory Committees has been made. We have also received aid from Dr. Mervin J. Kelly, Committee Chairman, who was the Chairman of the 1953 National Academy Committee and has since served as Chairman of the Statutory Visiting Committee of the Secretary of Commerce for the Bureau of Standards. The focus of our study has been the measurement of the Bureau's progress in implementing the recommendations of the 1953 study, evaluation of present strengths, and the formation of a judgment of the needs of the Bureau over the next several years in meeting its statutory obligations and in rendering the services to the nation that its rapidly expanding scientific and technical endeavors require.

"The Bureau's total full-time staff in June 1959 was 2,960. Some 1,500 were professional scientists or engineers. These were distributed by fields of specialization as follows: 525 physicists, 300 chemists, 375 engineers, 95 mathematicians, and 175 others. Two hundred seventy-five had obtained their Ph.D. degree, more than one hundred additional had academic training to that level. It is encouraging to note that the staff is now younger

[compared with 1955 figures—ed. note], there being a significant increase in the percentage of the staff in the 20- to 29-year age group with percentages in all older age groups, except the highest, having decreased.

"The grade with largest percentage of employees has advanced from GS–9 to GS–12 [compared with 1953 figures—ed. note]. At the same time, average salaries and salaries associated with each grade level have increased. The present average salary of the professional staff is approximately $8,500 per year.

"People are by far the most important resource of a scientific organization. The Bureau is no exception. For many years it has been staffed by an excellent group of scientists, highly qualified to work in a wide variety of fields. As a result, they have not only been able to perform effective work on measurements, but time and again have served the government by providing special talents to meet other pressing needs. Nowhere else in government does there appear to be an organization with such broad scientific outlook, coupled with such probable future long-term continuity of mission. For these reasons the Bureau represents an important and permanent scientific resource of government, and should be given the support warranted by so valuable an asset that insures the maintenance and enlargement of its scientific strength and effectiveness.

"The Bureau's management gives evidence of appreciating the importance of an educational program for maintaining staff competence. A program of graduate studies was initiated at the Bureau in 1908 and the Bureau gave formal recognition to the 50th Anniversary of this program in 1958. A substantial number of the present members of the Bureau's staff were assisted in obtaining advanced degrees through participation in this program. The courses provided in this educational program are recognized for advanced credit by most of the leading universities throughout the country and the majority of these courses are now listed in the catalogs of one or more of the universities in the Washington area. In some instances the courses are given by university staff members and in others by Bureau staff members.

"During the 1958–1959 school year there were approximately 32 courses offered in the Washington area with a total enrollment of about 500, half of which were from NBS. All of these 32 courses were offered out of hours.

"In addition, the Bureau conducts in-hours training courses entirely on its own in areas specifically related to job requirements. A total of 27 such courses was given in the past school year with approximately 800 enrollments, approximately half of which were from NBS and half from other agencies, including the Diamond Ordnance Fuze Laboratories of the Department of Defense that are housed in the Bureau's area. Similar programs, some of which are in cooperation with the University of Colorado, are provided at the Boulder Laboratories.

"In 1958 the Congress passed an employee training bill which has augmented significantly the Bureau's ability to provide specialized advanced training to selected staff members. It is now possible for the Bureau to send individuals for as much as a full academic year to educational institutions for intensive training. In addition, short-term training courses are possible. Although this authority was just made available in 1958, the Bureau has already made considerable use of this opportunity.

General Evaluation

"The Bureau of Standards is well administered. The Director and his staff have done an outstanding job of managing the limited resources in manpower and facilities to achieve a high level of effectiveness. The staff is of excellent scientific and engineering quality. Within the framework of government employment and compensation policies and practices, a remarkable job of recruitment and building of staff through educational programs and other means has been done.

"The development of accurate methods of physical measurement and of standards is of large importance to the commerce and industry of our country. Their needs for standards should be fully met. This the Bureau is not now doing only because of inadequate funds for its operation and for buildings and facilities. Within its resources, the Bureau's performance of its measurement standards function is excellent.

Summary of Principal Recommendations

"1. The measurement standards effort be expanded to provide adequate coverage of the Nation's standards needs. A rate of increase of effort of about 15 percent per year is suggested. This rate can be realized without noticeable loss of efficiency.

"2. The new facilities now planned for construction at the Gaithersburg, Maryland, site be provided at the earliest time possible.

"3. The existing mission statement for Radio Propagation and Standards should be issued as an Executive Order. A similar statement for Data Processing should be prepared, evaluated and, if found appropriate, given similar status.

"4. The Secretary should arrange for the establishment of committees of scientists and engineers to review the programs of the Cryogenics Engineering Laboratory, the Hydraulics Laboratory and the Building Technology Division to determine the

scope of the Bureau's responsibilities in these areas.

"5. Directly appropriated funds be obtained for all activities in the Measurements and Standards area that are of broad national interest. Transferred funds be employed only for activities of specific interest to the agency providing the funds.

"6. The Bureau be given a major increase in number of Public Law 313 positions. Its present request for 45 appears modest.

"7. The Technical Advisory Committees have a large potential value not yet fully realized. The Director continue his vigorous efforts to bring them to a higher level of effectiveness.

"8. The Director critically review all of the miscellaneous activities for other agencies such as testing, evaluation of materials and production, and report to the Statutory Advisory Committee the details and results of his analysis."

The 10-member Committee with Dr. Mervin J. Kelly, recently retired president of the Bell Laboratories, Chairman, was appointed in 1958 by Dr. Detlev W. Bronk, National Academy President, at the request of Commerce Secretary Sinclair Weeks "to evaluate the functions and operations of the Department of Commerce in relation to present national needs". Final examining work was completed in September 1959.

NEW STANDARD METAL-ORGANIC MATERIALS for analysis of petroleum products

A SET of 24 standard samples of metal-organic compounds [1] suitable for spectrographic and chemical analysis of petroleum products is now available from the Bureau. These stable oil-soluble substances—the result of 3 years of research and development conducted for the American Petroleum Institute—were selected from over 150 prospective compounds which had been synthesized and investigated [2] under the direction of Dr. H. S. Isbell, head of the organic chemistry laboratory.

For many years chemists in the petroleum industry have needed accurate standard samples for determining metals in petroleum products. From the spectrographic determination of the metals that accumulate in crankcase oils, engineers can judge engine wear and anticipate trouble prior to engine failure. Periodical analyses of oils from diesel locomotive engines are now made by several large railroads to detect faulty bearings. The method requires standard samples containing known quantities of the elements in question. Determination of metallic constituents in petroleum products is also highly important in refining processes and in the use and control of materials added to oils to improve lubricating properties.

Metal-organic standards containing the following elements are now available: Aluminum, barium, boron, cadmium, calcium, chromium, cobalt, copper, iron, lead, lithium, magnesium, manganese, mercury, nickel, phosphorus, potassium, silicon, silver, sodium, strontium, tin, vanadium, and zinc. All except six of these elements are incorporated in the form of the metal salt of cyclohexanebutyric acid. The other elements are dispensed as menthyl borate, tris (*O*-hydroxy-acetophenone) chromium-III, triphenyl phosphate, octaphenylcyclotetrasiloxane, dibutyltin bis (2-ethylhexoate), and bis (benzoylacetonate)-oxo-vanadium-IV. Spectrographic and chemical analyses for certification of these compounds were conducted under the direction of J. L. Hague and B. F. Scribner of the analytical and spectrochemical laboratories, respectively.

These selected compounds form stable solutions with lubricating oils. However, to increase the solubility of some of the samples, 2-ethyl-hexanoic acid and 6-methyl-2,4-heptanedione are used as additives. The standard samples are all compatible with each other so that blends containing various amounts of each can be prepared as needed.

For analysis of a petroleum product, accurately known amounts of the required metal-organic samples are added to a typical base material that is free of the elements provided in the samples. The resulting standard mixture is carried through all the steps involved in the analysis of the "unknown" oil. The amounts of the metallic impurities in the petroleum product are then determined by comparison with the results for the standard mixture.

The standard metal-organic materials, prepared in crystalline form for easy weighing and handling, may be purchased for $6.00 per 5 g of material. A certificate of analysis, plus directions for preparing a solution of the substance, is distributed with each sample. Orders should be directed to the Standard Sample Clerk, National Bureau of Standards, Washington 25, D.C.

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[1] *The National Bureau of Standards distributes over 600 different certified standard samples of chemicals, ores, ceramics, and metals. A complete listing of the standard materials issued by the Bureau is contained in NBS Circular 552 (3d edition),* Standard Materials, *which is available from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. (35 cents).*

[2] *A paper, describing the experimental procedures for preparing, purifying, and analyzing the metal-organic standard samples, is now being prepared.*

THE BUREAU has recently completed a 3-year experimental and theoretical study on free radicals, sponsored by the Department of Defense.[1] Scientists from industry joined with the NBS staff in this Free Radicals Research Program [2] to probe the secrets of short-lived atomic and molecular fragments. This centralized research activity not only revealed much about the production and nature of radicals but also paved the way for research in several new areas, including low-temperature chemistry and the photochemistry of solids. Other important results of the program are: A better understanding of low-temperature phenomena; improvements in low-temperature techniques;[3] and advances in spectroscopic instrumentation and in X-ray and electron diffraction techniques.

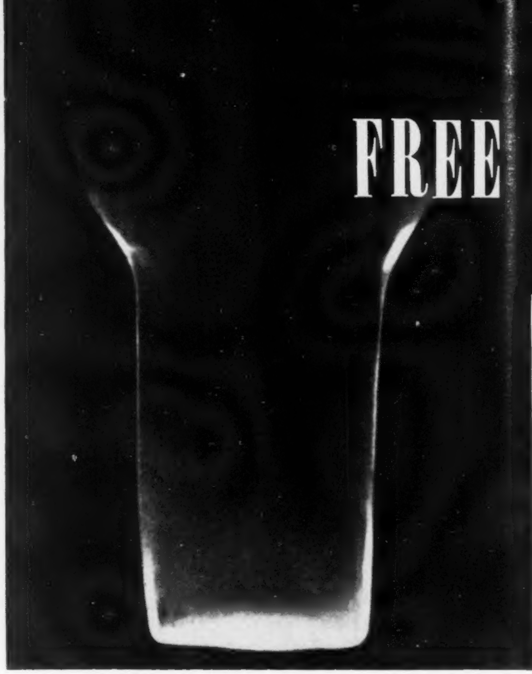
Although free radicals have been known to exist for over 50 years, only recently has research interest been focussed on their stabilization. Stabilized radicals can either be produced in a gas, which is then deposited as a condensed solid on a cold surface, or they can be produced, after deposition, in the condensed solid itself. In either case the excited species are frozen into immobility and sometimes display unusual phenomena such as bright glows, "flames", and flashes of light. When warmed up, they recombine actively, releasing stored energy.

It was the hope of trapping radicals in large quantities and storing them as a potential source of energy that sparked the request for the Bureau to undertake a comprehensive research program. However, from the start emphasis was placed on obtaining basic data and exploring various means of radical production rather than on solving a specific developmental problem. This basic-research approach has made the program an outstanding scientific contribution in spite of the finding that radical concentrations sufficient for practical use probably can not be obtained.

Production of Free Radicals

Of the several methods available for generating and trapping free radicals, the most extensively used at the Bureau was an original high-temperature discharge, low-temperature collection technique[4] developed by H. P. Broida and J. R. Pellam.[5] In this method, gases are passed through a high-frequency microwave discharge at 2,450 Mc/sec and then frozen at 4.2°K. The concentration of atoms and radicals produced in this way has been found to be relatively independent of the power applied to the resonator. In nitrogen, for example, magnetic susceptibility and electron spin resonance measurements indicate that the nitrogen atom concentration is about 0.1 percent at 4°K. When the concentration of atoms exceeds this value of 1 part in 1,000, the solid becomes unstable and explosions are caused by the recombination of atoms.

In another attempt at generating radicals in a gas, thermal dissociation was employed. One system, which was designed by L. E. Kuentzel,[6] has been used by J. T. Clarke,[7] J. J. Comeford, and J. H. Gould to study solids containing unstable molecular fragments. R. Klein[8] and M. D. Scheer employed another system in which hydrogen atoms, produced on a hot tungsten surface, diffuse into and react with cold solids to produce radicals.[9]



The glow of free radicals trapped in a matrix of frozen gas at liquid helium temperature.

Although it is possible to produce free radicals in a gas by photolysis (ultraviolet irradiation) or radiolysis (X- or γ-irradiation), these techniques were given no serious study. On the other hand, L. Wall, D. Brown, and R. E. Florin have found γ-ray radiolysis of the solid condensed from the gas to be a highly successful method of radical production, and E. Hörl[10] has employed electron bombardment extensively with results similar to those obtained in the gaseous discharge method.[11] The concentrations obtained by exposing condensed solids to ionizing radiations rarely exceed 1 part in 1,000.

A fairly large amount of work on the mechanisms of radical production has been based on studies of free radical spectra. Even before the Free Radicals Research Program was initiated, a semiquantitative interpretation of some spectroscopic features of the very complex light emission from nitrogen deposits was given by C. M. Herzfeld and H. P. Broida. It was suggested that, in the deposition of nitrogen passed through an electric discharge, N atoms are deposited, radiate, and then recombine to form excited N_2 molecules which also radiate. Some atomic features (alpha lines)

ISULTS OF RICALS RESEARCH

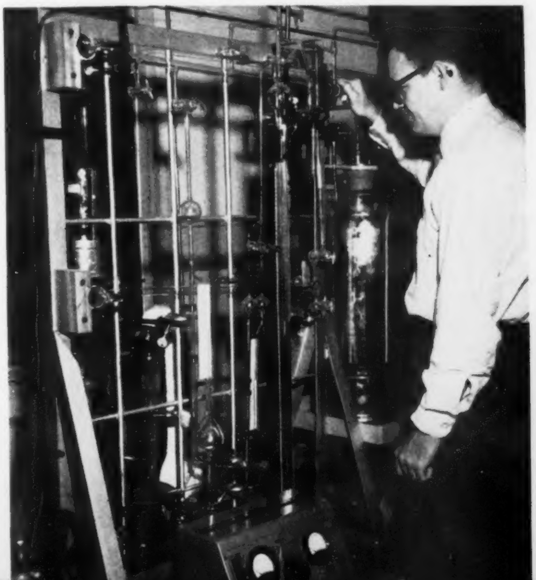
new data on molecular fragments

were attributed to a forbidden transition in N atoms; other atomic features (beta lines) were ascribed to transition between levels in O atoms; and the molecular features (bands) were assigned to a transition of the newly formed N_2 molecules.

It is now clear that the alpha lines do indeed arise from the transition proposed, although the situation is known to be vastly more complex than it was thought at first. The beta lines belong to transitions of O atoms and the bands to transitions of molecules. H. Goldberg and C. M. Herzfeld applied a crystal field analysis to the transition postulated for the beta atomic features, and have found reasonable agreement between experiment and theory.

Recently V. Griffing [12] has investigated the alpha lines from a different point of view, assuming that an N atom replaces an N_2 molecule in the crystal lattice, and then moves to readjust the lattice to minimize its energy. Several nonequivalent motions of this type can be found, which give rise to enough levels to explain the line group in question. So far the results obtained are only qualitative, but the approach is very promising.

O. S. Lutes uses a calorimeter to measure heat energy produced by the recombination of frozen free radicals.



Radical Deposition and Trapping

The condensation of a gas upon a cold surface is one of the least understood of the processes that occur during stabilization of radicals initially produced in the gas phase. When a molecule strikes a surface, it may rebound back into the gas after a single collision with the surface, it may become bound to a surface molecule for a time before it escapes back into the gas, or it may migrate at random over the surface and coalesce in a process of nucleation with other such particles and thus become a permanent part of the deposit. Measurements of the condensation of argon on a 4°K argon surface, by Bureau staff members F. A. Mauer and L. H. Bolz, and S. N. Foner of the Johns Hopkins Applied Physics Laboratory, indicated that 6 out of 10 atoms condensed, while Scheer found that the condensation of iodine on a 77°K glass surface occurred with nearly perfect efficiency. It is seen, therefore, that in addition to surface temperature, the material being deposited and the nature of the surface are of significance. In any case, a complete understanding of the stabilization of gaseous radicals in the solid state must be preceded by a more detailed insight into the deposition process. Although little experimental effort has been expended in this direction, a major part of the theoretical work has been devoted to explaining the mechanisms of radical deposition.

Several theories have been constructed to deal with the deposition problem. Two of these are statistical and study the number of ways in which radicals, molecules, and inert species can be laid down together. This approach, which employs various assumptions about the system, allows the

M. Peyron, guest worker in the Free Radicals Research Program, generates free radicals by an electric discharge technique. As radicals become trapped on a cold surface (here a refrigerated glass tube), they glow.

upper limits of radical concentrations to be estimated.

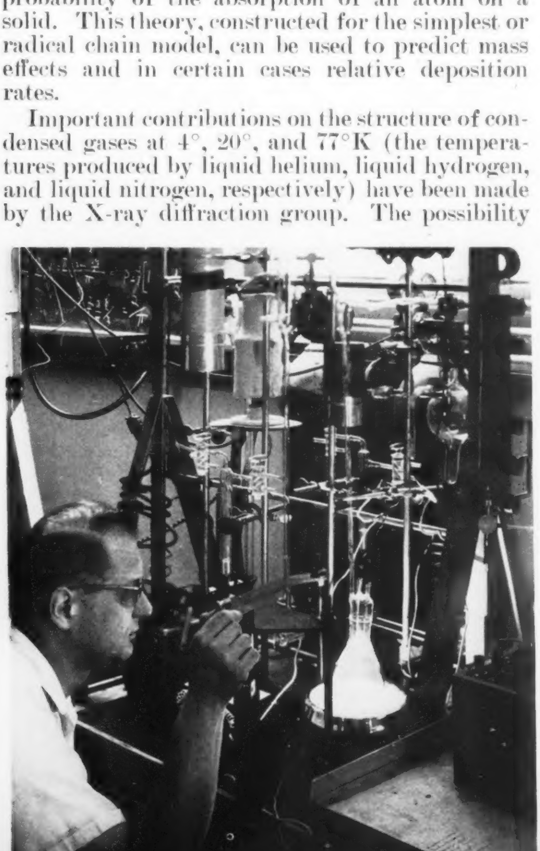
One such theory was developed by J. L. Jackson and E. W. Montroll,[13] who considered that radicals form chains. They studied three cases in detail: the deposition of radical chains, the deposition of linear chains containing radicals and molecules randomly distributed, and three-dimensional condensates obtained from a gas containing radicals only and from a gas containing radicals and molecules.

A second statistical theory was constructed by S. Golden,[14] who postulated five 3-dimensional condensation models: A densely packed model in which all sites in the solid are filled by a radical or a molecule; a diffusely packed model in which all species condense one to a site and then recombine where possible; a surface model in which each condensed layer comes to a stable state before the next layer is deposited; a nonideal model in which species may evaporate from a surface; and a clustering model which assumes that clusters form in the gas and are deposited as such.

Although the statistical theories have the advantage of being logically straightforward, they ignore energy considerations and diffusion and are therefore likely to give high estimates of trapped radical concentrations.

An attempt to approach the problem by studying the detailed mechanisms involved in deposition was made by R. Zwanzig who calculated the probability of the absorption of an atom on a solid. This theory, constructed for the simplest or radical chain model, can be used to predict mass effects and in certain cases relative deposition rates.

Important contributions on the structure of condensed gases at 4°, 20°, and 77°K (the temperatures produced by liquid helium, liquid hydrogen, and liquid nitrogen, respectively) have been made by the X-ray diffraction group. The possibility of obtaining information about the number and type of crystallographic sites available for free radical trapping is an attractive feature of this type of investigation. The materials that have been studied in some detail are argon, nitrogen, oxygen, water, ammonia, diborane, hydrazoic acid, and various alcohols. L. H. Bolz, M. E. Boyd, F. H. Mauer, and H. S. Peiser found that, in general, slow rates of gaseous deposition result in solids with a considerably less ordered structure than that obtained with rapid deposition rates. The use of an electric discharge in the gas prior to deposition increases order in solids, and heating amorphous solids causes an annealing to the stable crystalline configuration. Part of the released energy observed in the warmup of excited solids must be attributed to these crystallographic changes.



Some interesting results were obtained by Hörl in an electron diffraction study of thin solid nitrogen films at 4°K and 20°K. For extremely thin films at 4°K, the observed diffraction patterns indicate the presence of randomly oriented fine crystallites. As the amount of deposited material is increased, larger crystals are formed.

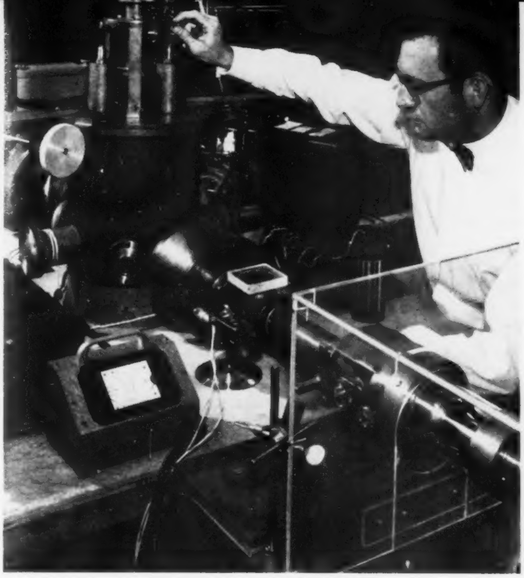
A number of significant facts about trapping forces have been discovered through spectroscopic studies. It has become fairly clear that the trapping of atoms in molecular lattices is a complicated phenomenon. For example, in the cases of N trapped in N_2 and O trapped in N_2, loose complex formation with molecules occurs. Both theoretical and experimental evidence lead to this conclusion.

Trapping forces have also been investigated by Jackson and B. Castle, who attempted to discover the existence of positions in or on an N_2 lattice where an N_3 molecule could rotate, either freely or in a hindered manner. They calculated the energy levels of molecules on the surface of the crystal, and also studied the long-range interaction potential between N and N_2. Although experimental evidence for such behavior exists in the spectral lines, the present results are so far inconclusive.

Characterization of Stabilized Radicals

Once it has been established that radicals are present in a cold solid, the first question to arise is that of concentration stability. Wall, Brown, and Florin have performed several experiments which yielded information on this point. For example, electron spin resonance spectrometer measurements on nitrogen atoms generated in solid nitrogen by γ-irradiation showed no decrease in signal strength after the solid had been stored at 4° K for 20 hr. On the other hand, a similar

In studies of low-temperature reactions, M. D. Scheer produces free radicals by bombarding a solid with hydrogen atoms.



E. Hörl generates free radicals by electron bombardment.

investigation on hydrogen showed a 50-percent decrease in concentration.

Trapped radicals disappear by at least two distinct mechanisms. One type of flash seems to propagate so rapidly that it appears to cover an area of several square millimeters instantaneously. A second type of flash—called a "cold frame"—propagates so slowly that the movement of a definite front can be discerned. Recent thermal and magnetic studies by B. J. Fontana [15] indicate that these flashes are intimately connected with some critical concentration of trapped radicals. This finding has led Jackson to work out a comprehensive treatment of stability in trapped radical systems and of the ways in which unstable systems disappear.

This work consists of three major parts: The first part uses qualitative arguments to obtain a simple expression for a critical concentration, n_c, of trapped radicals, which, if exceeded, gives unstable systems that decay explosively; the second part consists of a detailed analysis of discrete sequences of radical reactions, with an analysis of the circumstances under which steady states can exist; and the third part involves a study of the partial differential equations for the average values of concentrations. Remarkably, all three treatments give the same criterion for stability.

In addition to the techniques already mentioned—electron spin resonance and optical spectroscopy—the Bureau has employed mass spectrometry in studying free radicals. This method was used by J. T. Herron, J. L. Franklin,[16] P. Bradt, and V. A. Dibeler to study gaseous recombination kinetics, and to investigate the reactions of atoms in the gas phase. They also made a mass spectrometric determination of the N-atom concentration in the afterglow region of nitrogen excited by the electric discharge method. This number, which is between 1 and 2 percent, provides an upper limit to the concentration of atoms in the condensed nitrogen.

H. P. Broida, O. S. Lutes,[17] and G. J. Minkoff,[18] F. Scherber,[19] and J. Gallagher have used calorimeters to measure the energy liberated by various discharged gases condensed at 4°K. Although results indicate that as much as several percent of trapped energetic species are present in the solids produced in this fashion, this estimate is thought to be too high.

Other measurements on gases condensed at 4°K included determinations of the index of refraction by J. Kruger; the thermal conductivity by R. J. Corruccini, H. Roder, J. T. Clarke, A. Chatenever,[20] and R. Gorden; and the dielectric constant by N. L. Brown. All results were consistent with the finding that radical concentrations generally are very low.

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[1] *A complete discussion of the Bureau's work on free radicals, both experimental and theoretical, is included, along with references to significant publications, in NBS Monograph* **12,** Stabilization of free radicals at low temperatures (*in press*).

[2] Free radicals research program, *NBS Tech. News Bul.* **41,** *1* (*1957*).

[3] Providing liquid refrigerants to research workers, *NBS Tech. News Bul.* **43,** *146* (*1959*); Glass dewars for trapped radical studies, *Tech. News Bul.* **43,** *40* (*1959*).

[4] Low temperature storage of free radicals, *NBS Tech. Bul.* **40,** *112* (*1956*). Patent for free radical stabilization, *NBS Tech. News Bul.* **43,** *175* (*1959*).

[5] *Now on the faculty of the California Institute of Technology.*

[6] *Guest worker from the Wyandotte Chemicals Corp.*

[7] *Guest worker from the Ethyl Corp.*

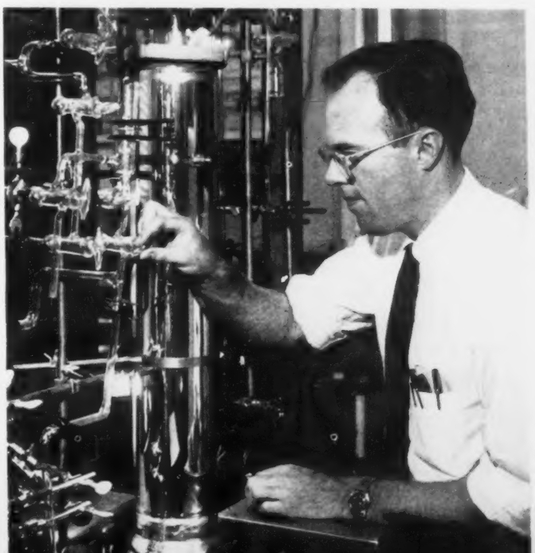
[8] *Guest worker from the Olin-Mathieson Chemical Corp.*

[9] Studies of reactions with low activation energies, *NBS Tech. News Bul.* **43,** *206* (*1959*).

[10] *Now at Howard University.*

[11] Spectroscopic studies of trapped radicals, *NBS Tech. News Bul.* **43,** *164* (*1959*).

J. J. Comeford uses a Dewar specially designed for studying solids containing free radicals.



[12] *Guest worker from the Catholic University of America.*

[13] *Guest worker from the University of Maryland.*

[14] *Guest worker from Brandeis University.*

[15] *Guest worker from the California Research Corp.*

[16] *Guest worker from Humble Oil and Refining Co.*

[17] *Now with the Minneapolis-Honeywell Regulator Co.*

[18] *Guest worker from the Imperial College of Science and Technology (London).*

[19] *Guest worker from the Union Carbide Corp.*

[20] *Guest worker from the Sinclair Research Laboratories.*

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The photolysis of ammonia in a solid matrix at low temperature, by O. Schnepp and K. Dressler, J. Chem. Phys. (in press).

Electrical discharge induced luminescence of solids at low temperatures, by L. J. Schoen and R. Rebbert, J. Mol. Spect. **3**, 417 (1959).

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Isotope exchange processes in solid nitrogen under electron bombardment, by R. Klein and E. Hörl, J. Chem. Phys. **32**, 307 (1960).

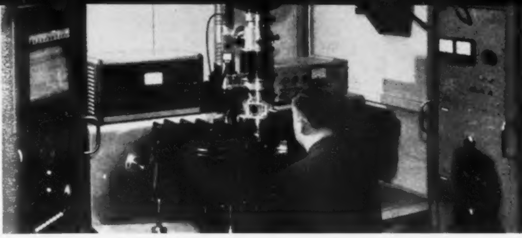
METALLURGICAL CONFERENCE

THE BUREAU held a 1-day Metallurgical Conference at its Washington laboratories on March 31. In attendance were more than 100 representatives of industrial, academic, and government laboratories having major programs in metallurgy and metal physics. Five scientific papers were given during a technical session, which was followed by an "open house" at the metallurgy laboratories for the inspection of new instruments.

Dr. James I. Hoffman (center), chief of the Bureau's metallurgy laboratories, greets Samuel Epstein (left) of Bethlehem Steel Corp., and Col. Alexander I. Krynitsky (right), retired Bureau staff member, at the Bureau's metallurgical conference. Mr. Epstein and Col. Krynitsky have been attending these conferences, held almost every year at the Bureau, since 1918.

Purpose of the conference was to present results of recent Bureau research in areas of current interest. Since 1913 the Bureau has been conducting research on the structure and properties of metals. During this time, conferences have been held almost every year to acquaint metallurgists and engineers with the progress of the work, and to explain newly developed methods for studying metals and alloys.

The conference was opened by Dr. Allen V. Astin, NBS Director, who spoke of the need for metals research throughout the Nation. Dr. James I. Hoffman, chief of the metallurgy laboratories, then outlined the experimental work now going on at the Bureau in metal physics, physical metallurgy, and corrosion. Dr. Hoffman concluded his talk by introducing two conferees, Samuel Epstein of the Bethlehem Steel Corp., and Col. Alexander I. Krynitsky, both of whom have



J. R. Cuthill observes an alloy specimen with the electron probe microanalyzer he designed. The instrument, now nearing completion, was on display at the NBS metallurgical conference. Fluorescent X-ray emission characteristic of the elements in the specimen is analyzed spectrometrically.

attended all of the metallurgical conferences over the past 42 years. Mr. Epstein was on the Bureau staff from 1918 until 1929, and Col. Krynitsky from 1918 until his retirement in 1950.

Dr. L. M. Kushner, head of the metal physics laboratory, presided at the technical session. The first paper was given by M. R. Meyerson, who reviewed advances made in the past year in developing materials suitable for extremely precise gage blocks. This work was undertaken to provide gage blocks that may be certified to one ten-millionth of an inch. Recent experiments indicate that a nitrided stainless steel has many of the desired characteristics and closely approaches the dimensional stability needed in ultraprecise gage blocks.

Dr. J. R. Manning explained some of the atomic mechanisms of diffusion in metals. When diffusion occurs by a vacancy mechanism, a diffusing atom does not make a succession of random jumps; rather, a correlation exists between the directions of successive jumps. He showed how this correlation affects both tracer diffusion experiments performed by physicists, and chemical diffusion experiments by metallurgists.

Recent results obtained in studies of stress-corrosion cracking of both ferrous and non-ferrous metals were presented by H. L. Logan. He indicated that during crack propagation the strain rate at the tips of cracks is sufficiently high to prevent the reforming of protective films. Thus an electrochemical reaction, in which the crack tips are the anodes and the crack sides the cathodes, is important in the propagation of stress-corrosion cracks.

The application of nuclear magnetic resonance experiments to metals was described by Dr. L. H. Bennett. He gave basic principles, and then related results obtained on the nuclear resonance of tantalum both in the metallic state and in a nonmetallic compound, and in measurements on a series of lead-indium alloys.

The final speaker, Dr. C. J. Newton, reported on the cyclic stressing of polycrystalline cartridge brass specimens. His experiments showed that intragranular misorientations, produced by the initial deformation and measured with an X-ray goniometer, can be reduced by straining the specimens in the reverse direction. The yield stresses measured after several deformation cycles were discussed in the light of current theories.

Equipment installed in the metallurgy laboratories since the last conference was inspected by the visitors at the conclusion of the technical session. Particular interest was displayed in an electron probe microanalyzer, designed by Dr. J. R. Cuthill, which promises to be useful in determining phase diagrams of alloys and in studying diffusion processes. Another instrument attracting special notice was an electron-beam zone melter, developed by Dr. George A. Moore, which will be employed in work on high-purity and standard metals.

1960 Cryogenic Engineering Conference

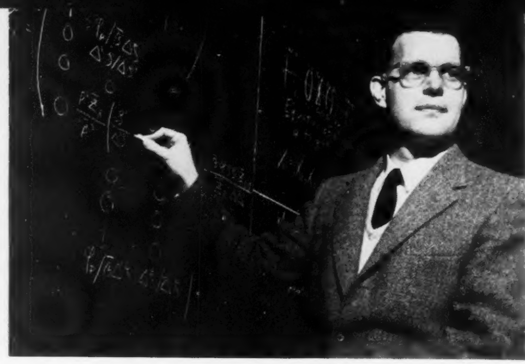
THE 1960 Cryogenic Engineering Conference, co-sponsored by the University of Colorado and the Bureau, will be held in Boulder, Colorado on August 23, 24, and 25.

The field of cryogenic engineering is growing rapidly, as larger and larger quantities of the liquefied gases, such as hydrogen, oxygen, nitrogen, and helium are being used by industry and national defense, and for laboratory research. This has necessitated the design and construction of safe, large, and more efficient equipment for producing and using them. Unusual engineering problems are encountered because of the marked differences in the behavior of materials at very low temperatures as compared with ordinary temperatures.

Subjects which will be discussed at the conference include liquefaction cycles, purification of gases, gas separation, distillation, heat transfer, catalysis, fluid flow, absorption, hydrogen and LOX production, cryogenic fuels, oxidants, pressurants, missile problems, mechanical and thermal properties, vacuum insulation, powder insulation, super insulation, safety, friction studies, vapor-liquid equilibria, liquid level devices, probes, pumps, bearings, transfer lines, dewars, cryostats, temperature and pressure measuring devices, expansion engines and turbines, heat exchangers, regenerators, high-energy and nuclear applications, bubble chambers, and cognate topics.

Information regarding registration and reservations may be obtained from K. D. Timmerhaus of the Chemical Engineering Department, University of Colorado, Boulder, Colorado, who is Secretary of the Conference Committee.

Lens Design Problems Solved by Matrix Algebra



O. N. Stavroudis explains his method for using matrix algebra in solving lens design problems. On the blackboard is a system of partial differential equations, defining optical image formation, written in the form of 4-by-4 matrices.

WORKING under the sponsorship of the U.S. Air Force, the Bureau has developed an improved method that employs matrix algebra in designing optical lenses.[1] In this method, devised by O. N. Stavroudis of the optical instruments laboratory, a system of partial differential equations defining optical image formation is written in the form of 4-by-4 matrices. The form of these matrices suggests a new method of designing lenses.

The greatest stumbling block to the lens designer is the difficulty of correlating the aberrations of an optical system with such parameters as the curvatures, separations, and thicknesses of the lenses and the indices of refraction. Thus the design of a lens is ordinarily a trial-and-error process, where values are assigned to the parameters, rays are traced, and the results are used to assign values representing the aberrations.

In the improved method, the aberrations of a lens can be defined in such a way that their relationship to the parameters is explicit. Here the elements of the 4-by-4 matrix represent the total aberrations of an optical system. If X is such a matrix, and the optical system represented by X has two components, then $X=X_1X_2$, where X_1 and X_2 are the X matrices for each of the components. Each X can be further split into additional components. This splitting can go on until the ultimate components are reached; these are (a) a refracting surface with which is associated a "refraction matrix X_R," and (b) the space between two refracting surfaces with which is associated a "transfer matrix X_T."

Most optical systems consist of spherical surfaces separated by homogeneous isotropic media (glass and air). In this case X_T depends on two parameters, the distance between two successive surfaces and the index of refraction of the medium between them, and X_R depends on the curvature of the surface and the index of refraction of the two media on either side of the surface. Thus the X matrix describing the total monochromatic geometric aberration of an optical system can be represented by a product of X_R and X_T matrices.

This principle can be used to set up a matrix equation whose solution is the design of a lens meeting certain prescribed requirements. Describing these requirements in terms of an X matrix and then setting it equal to a product of X_R-type and X_T-type matrices results in the required equation. Such an equation in 4-by-4 matrices is equivalent to a system of 16 simultaneous scalar equations. By assuming rotational symmetry and by making use of the system of differential equations mentioned above that apply to all optical systems, the complexity of the equations of condition may be reduced.

[1] *For further technical information, see* Lens design: a new approach, *by O. N. Stavroudis, J. Research NBS* **63B**, *No. 1, p. 31 (July–September 1959). Part of this work was performed at the Imperial College of Science and Technology and included in a thesis submitted to the University of London.*

Publications of the National Bureau of Standards

Periodicals

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Section D. Radio Propagation. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75.

Section A. Physics and Chemistry, Volume **64A**, *No. 3, May–June 1960.*

Phase shift effects in Farbry-Perot interferometry, C. J. Koester.

Infrared absorption spectrum of methane from 2,470 to 3,200 cm^{-1}, E. K. Plyler, E. D. Tidwell, and L. R. Blaine.

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Radial distribution study of vitreous barium borate, A. Bienenstock, A. S. Posner, and S. Block.

Separation and determination of small quantities of aluminum in steel, B. B. Bendigo and R. K. Bell.

Conformations of the pyranoid sugars. II. Infrared absorption spectra of some aldopyranosides, R. S. Tipson and H. S. Isbell.

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Non-self-adjoint boundary value problems in ordinary

differential equations, W. Greub and W. C. Rheinboldt.
Criteria for the existence and equioscillation of best Tchebycheff approximations, J. R. Rice.
Note on the solution of Riccati's differential equation, H. H. Howe.
On a generalization of the index notation for absolute tensors of arbitrary order, E. H. Brown.
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Half-round inductive obstacles in rectangular waveguide, D. M. Kerns.

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Apparatus for the measurement of the normal spectral emissivity in the infrared, A. G. Maki, R. Stair, and R. G. Johnston.
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The functional synthesis of linear plots, J. P. Vinti and R. F. Dressler.
Radiation field from a rectangular source, J. H. Hubbell, R. L. Bach, and J. C. Lamkin.
Microwave attenuation measurements with accuracies from 0.0001 to 0.06 decibel over a range of 0.01 to 50 decibels, G. F. Engen and R. W. Beatty.
Effect of oleophobic films on metal fatigue, H. E. Frankel, J. A. Bennett, and W. L. Holshouser.
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Section D. Radio Propagation, Volume **64D**, *No. 3, May–June 1960.*

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A theory of wavelength dependence in ultrahigh frequency transhorizon propagation based on meterological considerations, R. Bolgiano, Jr.
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Trade-wind inversion as a transoceanic duct, M. Katzin, H. Pezzner, B. Y.-C. Koo, J. V. Larson, and J. C. Katzin.
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On the mode theory of very-low-frequency propagation in the presence of a transverse magnetic field, D. D. Crombie.
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Focusing, defocusing, and refraction in a circularly stratified atmosphere, K. Toman.
Response of a loaded electric dipole in an imperfectly conducting cylinder of finite length, C. W. Harrison, Jr., and R. W. P. King.
Impedance characteristics of a uniform current loop having a spherical core, S. Adachi.

Technical News Bulletin, Volume **44**, *No. 5, May 1960. 15 cents.* Annual subscription: $1.50, 75 cents additional for foreign mailing.

Basic Radio Propagation Predictions for August 1960. Three months in advance. CRPL–D189, issued May 1960. 15 cents. Annual subscription $1.50, 50 cents additional for foreign mailing.

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FREDERICK H. MUELLER, *Secretary*
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A. V. ASTIN, *Director*

June 1960 **Issued Monthly** **Vol. 44, No. 6**

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Contents

Publications (*Continued*)

Atmospheric tides and ionospheric electrodynamics, M. L. White. J. Geophys. Research **65**, 153 (1960).

Some evidence for structural anomalies in pure cristobalite, R. F. Walker, S. J. Schneider, and R. S. Roth. J. Am. Ceram. Soc. **42**, No. 12, 642 (1959).

La Recherche sur les radicaux libres au National Bureau of Standards, H. P. Broida, J. Chim. Phys. **56**, No. 2392, 813 (1959).

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Patents

(The following U.S. Patents have recently been granted on NBS inventions and, except as noted, are assigned to the United States of America as represented by the Secretary of Commerce.)

No. 2,920,157 January 5, 1960. Inertia switch. Jacob Rabinow and William B. McLean (Navy).

No. 2,920,568 January 12, 1960. Turbo-generator system. Jacob Rabinow (Army).

No. 2,920,984 January 12, 1960. Paper coating composition, paper coated therewith, and method of improving the strength of paper. John T. Moynihan.

No. 2,921,518 January 19, 1960. Explosive ordnance construction. Robert D. Huntoon (Navy).

No. 2,921,522 January 19, 1960. Multiple detonator operation. Maurice Apstein (Army).

No. 2,921,524 January 19, 1960. Fuze safety device. Jacob Rabinow (Navy).

No. 2,921,549 January 19, 1960. Holding fixture for spin-forming blanks. Earl L. Schwenk.

No. 2,922,121 January 19, 1960. Gated beam tube relaxation oscillator. Carroll E. Tschiegg.

No. 2,922,963 January 26, 1960. Adjustable waveguide termination. Robert W. Beatty.

No. 2,923,209 February 2, 1960. Tool shaping machine. Carl E. Pelander (Army).

No. 2,924,562 February 9, 1960. Method of free radical production and stabilization. Sidney Golden.

No. 2,925,587 February 16, 1960. Magnetic drum memory for electronic computers. Ragnar Thorensen, William R. Arsenault, Biagio F. Ambrosio.

No. 2,926,611 March 1, 1960. Circuit controlling means. Wilbur S. Hinman, Jr. (Navy).

No. 2,927,138 March 1, 1960. Method for the preparation of hexafluorobenzene. Leo A. Wall, Max Hellman, and Walter J. Pummer.

No. 2,927,213 March 1, 1960. Electronic control circuit. Thomas M. Marion and John F. Streib (Navy).

No. 2,927,214 March 1, 1960. Signal translating system. Joseph G. Hoffman (Navy).

No. 2,928,347 March 15, 1960. Inertia arming switch. William B. McLean (Navy).

No. 2,928,937 March 15, 1960. Electroluminescent microwave receiver. George G. Harman, Jr.

No. 2,929,027 March 15, 1960. Amplifier including hum elimination control means. Finley L. Cooke (Navy).

No. 2,929,742 March 22, 1960. Electroless deposition of nickel. Clara H. deMinjer and Abner Brenner.

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